

EXPRESS MAIL LABEL # EL822430026US

JC07 Rec'd PCT/PTO 1 4 FEB 2002

Form PTO-1390 REV 10-94)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEYS DOCKET NUMBER RN99101
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/elected office (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (IF KNOWN, SEE 3737 CFR 1.5) 10/049575 NOT APPLICABLE
INTERNATIONAL APPLICATION NO. PCT/FR00/02325	INTERNATIONAL FILING DATE August 16, 2000	PRIORITY DATE CLAIMED August 17, 1999	
TITLE OF INVENTION USE OF POLYOXYPROPYLENE/POLYOXYETHYLENE TERPENE COMPOUNDS AS DEGREASING AGENTS FOR HARD SURFACES			
APPLICANT (S) FOR DO/EO/US: Jean-Luc JOYE and Jean-Guy LE HELLOCO			
Applicant herewith submits the United States Designated/Elected Office (DO/EO/US) the following items and other information: <ol style="list-style-type: none">1 <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U S C 3712 <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U S C 3713 <input checked="" type="checkbox"/> This express request to begin national examination procedures (35 U S C 371) (f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U S C 371 (b) and PCT Articles 22 and 39(1)4 <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date5 <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U S C 371(c)(2))<ol style="list-style-type: none">a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US)6 <input checked="" type="checkbox"/> A translation of the International Application into English (35 U S C 371(c)(2)).7 <input checked="" type="checkbox"/> A copy of the International Search Report (PCT/ISA/210)8 <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U S C 371(c)(2)).<ol style="list-style-type: none">a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the Internationalb. <input type="checkbox"/> have been transmitted by the International Bureauc. <input type="checkbox"/> have not been made, however the time limit for making such amendments has NOT expired.d. <input checked="" type="checkbox"/> have not been made and will not be made.9 <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U S C 371(c)(3)).10 <input checked="" type="checkbox"/> A oath or declaration of the inventor(s) (35 U S C 371(c)(4)). (unsigned)11 <input checked="" type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409).12 <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U S C 371(c)(5)). Items 13 to 18 below concern document(s) or information included: <ol style="list-style-type: none">13 <input checked="" type="checkbox"/> An information Disclosure Statement under 37 CFR 1 97 and 1 9814 <input checked="" type="checkbox"/> An assignment document for recording A separate cover sheet in compliance with 37 CFR 3 28 and 3 31 is included15 <input checked="" type="checkbox"/> A FIRST preliminary amendment16 <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment17 <input type="checkbox"/> A substitute specification.18 <input type="checkbox"/> A change of power of attorney and/or address letter19 <input checked="" type="checkbox"/> Certificate of Mailing by Express Mail20 <input checked="" type="checkbox"/> Other items or information Forms PCT/IB/332, PCT/ISA/ 210 (English & French Version), PCT/IPEA/ 409 (English & French Version), PCT/FR00/02325 as published			

US APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.5) <div style="font-size: 2em; font-weight: bold; margin-top: 10px;">10/049575</div>	International Application No. PCT/FR00/02325	ATTORNEY DOCKET NUMBER RN99101
		CALCULATIONS PTO ONLY
21 The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1,482) nor International search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO..... \$1,040.00 <input checked="" type="checkbox"/> International preliminary examination fee not paid to USPTO but International Search Report prepared by the EPO or JPO..... \$ 890.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1,482) not paid to USPTO but international search (37 CFR 1,445 (a)(2)) fee paid to USPTO..... \$ 740.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1,482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$ 710.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1,482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$ 100.00 <div style="text-align: center; font-weight: bold;">ENTER APPROPRIATE BASIC FEE AMOUNT =</div>		<div style="border: 1px solid black; padding: 5px; margin-bottom: 5px;">\$ 890.00</div> <div style="border: 1px solid black; padding: 5px;">\$ 0.00</div>
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1,492(e)).		
CLAIMS	NUMBER FILED	NUMBER EXTRA
Total Claims	18-20=	0
Independent Claims	1-3=	0
MULTIPLE DEPENDENT CLAIMS(S) (if applicable) N/A		<input type="checkbox"/>
TOTAL OF ABOVE CALCULATIONS =		\$ 890.00
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28)		\$ 0.00
SUBTOTAL =		\$ 890.00
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1,492 (f)).		\$ 0.00
TOTAL NATIONAL FEE =		\$ 890.00
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property + <input type="checkbox"/>		\$ 0.00
TOTAL FEES ENCLOSED =		\$ 890.00
		Amount to be refunded \$
		charged \$
a <input type="checkbox"/> A check in the amount of \$_____ to cover the above fees is enclosed b <input checked="" type="checkbox"/> Please charge my Deposit Account No. 18-1169 in the amount of \$ 890.00 to cover the above fees. c <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 18-1169 A duplicate copy of this sheet is enclosed.		
NOTE: Where an appropriate time limit under 37 CFR 1,494 or 1,495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.		
<div style="display: flex; justify-content: space-between;"> <div style="width: 40%;"> SEND ALL CORRESPONDENCE TO: John A. SHEDDEN RHODIA INC. 259 Prospect Plains Road CN 7500 Cranbury, NJ 08512 </div> <div style="width: 40%; text-align: center;"> SIGNATURE <hr style="width: 100%;"/> JOHN A. SHEDDEN NAME <div style="border-bottom: 1px solid black; width: 100px; margin: 0 auto;"></div> 25,644 </div> </div> <div style="display: flex; justify-content: space-between; margin-top: 20px;"> <div style="width: 40%;"> February 13, 2002 DATE </div> <div style="width: 40%; text-align: center;"> REGISTRATION NUMBER </div> </div>		

Express Mail Label #: EL 822430026 US

Case RN99101

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the Application of: Jean-Luc JOYE, and Jean-Guy LE HELLOCO

National Phase of PCT/FR00/02325

Examiner: N/A

International Filing Date : August 16, 2000

Serial No: To be assigned

Art Unit: N/A

Filing Date: To be assigned

For: USE OF POLYOXYPROPYLEN/POLYOXYETHYLENE TERPENE
COMPOUNDS AS DEGREASING AGENTS FOR HARD SURFACESCommissioner for Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Dear Sir:

Prior to calculation of filing fee, please enter the following amendment in the
specification and claims:

In the Specification:

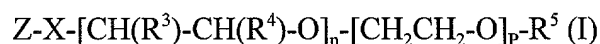
Page 1, just after the title, please add the new following paragraph:

This application is an application under 35 U.S.C. Section 371 of International
Application Number PCT/FR00/02325 filed on August 16, 2000.

In the Claims:

Please cancel claims 1-13 and replace them with the following new claims 14-31:

14. (New) A process for degreasing or cleaning a hard surface, comprising the step of
using an aqueous medium comprising at least one compound employed in a
concentration of from 0.01 to 10 g/l, having the following formula (I):



wherein:

- Z represents a bicyclo[a,b,c]heptenyl or bicyclo[a,b,c]heptyl group, wherein:

$$a + b + c = 5,$$

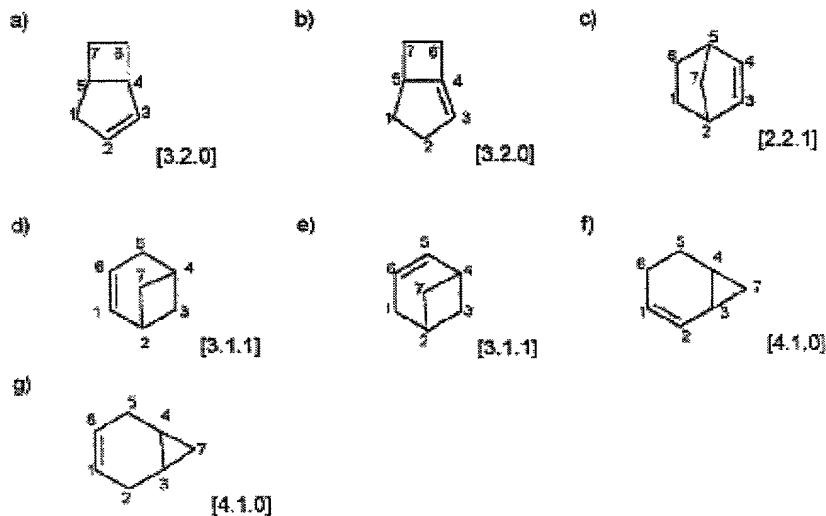
$$a = 2, a=3, \text{ or } a=4,$$

$$b = 2 \text{ or } b=1, \text{ and}$$

$$c = 0 \text{ or } c=1,$$

the bicyclo[a,b,c]heptenyl or bicyclo[a,b,c]heptyl group being optionally substituted by at least one C₁-C₆ alkyl group,

Z being selected from the group consisting of the groups of the following formulae a) to g), and the groups of the following formulae a) to g) minus the double bond:



- X represents -CH₂-C(R¹)(R²)-O- or -O-CH(R^{'1})-CH(R^{'2})-O-, wherein:

- R¹, R², R^{'1} and R^{'2}, which are identical or different, represent hydrogen, or a linear, branched or cyclic, saturated or unsaturated C₁-C₂₂ hydrocarbon group,

- R^3 and R^4 , which are identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1-C_{22} hydrocarbon group, provided that at least one of groups R^3 or R^4 is other than hydrogen,
- R^5 represents hydrogen, a linear, branched or cyclic, saturated or unsaturated, aromatic or non-aromatic C_1-C_{22} hydrocarbon group, which may be substituted, or a group selected from the group consisting of the following groups:

- SO_3M

- $OPO_3(M)_2$

-(CH_2)_r-COOM, and

-(CH_2)_z- SO_3M ,

wherein :

- M represents hydrogen, an alkali metal or an ammonium function $N(R)_4^+$, wherein R, which is identical or different, represents hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1-C_{22} hydrocarbon group, optionally hydroxylated,
- r is from 1 to 6, and
- z is from 1 to 6;
- n is an integer or a fractional number from 3 to 5 inclusive, and
- p is an integer or a fractional number from 6 to 10, limits excluded.

15. (New) A process according to claim 14, wherein the hard surface is a metal surface.

16. (New) A process according to claim 14, wherein R^1 , R^2 , R'^1 and R'^2 , which are identical or different, represent hydrogen, or a linear, branched or cyclic, saturated or unsaturated C_1 - C_6 hydrocarbon group.
17. (New) A process according to claim 14, wherein n is equal to 3.
18. (New) A process according to claim 14, wherein p is from 6.2 to 7, limits included.
19. (New) A process according to claim 18, wherein p is from 6.3 to 7, limits included.
20. (New) A process according to claim 19, wherein n is from 4 to 5.
21. (New) A process according to claim 14, wherein p is from 7 inclusive to 10 exclusive.
22. (New) A process according to claim 21, wherein p is from 8 inclusive to 10 exclusive.
23. (New) A process according to claim 14, wherein group Z is substituted on at least one of carbon atom by two C_1 - C_6 alkyl groups.
24. (New) A process according to claim 14, wherein X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ and Z is selected from the group consisting of the groups of formulae c) to g).
25. (New) A process according to claim 24, wherein Z is selected from the group consisting of the groups of formulae d) and e).
26. (New) A process according to claim 14, wherein X represents $-\text{O}-\text{CH}(\text{R}'^1)-\text{C}(\text{R}'^2)-\text{O}-$ and Z is a group having a backbone of formula c), without a double bond.
27. (New) A process according to claim 26, wherein Z is substituted by a C_1 - C_6 alkyl group.

28. (New) A process according to claim 27, wherein the C₁-C₆ alkyl group is a methyl group on carbon 2 or carbon 5 of the backbone of formula c).
29. (New) A process according to claim 14, wherein the hard surface is a metal plate, and the concentration of compound is from 0.01 to 5 g/l.
30. (New) A process according to claim 14, the hard surface is a platform, and the concentration of compound is in the range from 0.01 to 10 g/l.
31. (New) A process according to claim 14, wherein the hard surface is an oil production well, and the concentration of compound is from 0.01 to 5 g/l.

REMARKS

The preliminary amendments are filed to comply with the claims structure and wording according to the United States Patent law. It is asserted that these amendments do not add new matter. Support for these amendments can be found in the specification and claims as originally filed.

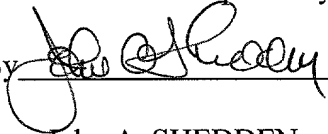
New claims find basis as mentioned in the chart below:

<u>New Claims</u>	Basis	
	In the claims as filed	In the specification
14	1	Page 10, line 13-14
15	1	
16	1	
17	2	
18	3	
19	3	
20	4	
21	5	
22	5	
23	6	
24	7	
25	8	
26	9	
27	10	
28	10	
29	11	
30	12	
31	13	

Entry of these amendments is respectfully requested.

February 13, 2002
Rhodia Inc.
259 Prospect Plains Road
CN7500
Cranbury, NJ 08512

Respectfully submitted,

By 
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USE OF POLYOXYPROPYLENE/POLYOXYETHYLENE TERPENE COMPOUNDS
AS DEGREASING AGENTS FOR HARD SURFACES

The present invention relates to the use of terpene compounds comprising oxypropylene and oxyethylene units as agents for degreasing/cleaning hard surfaces, more particularly metal surfaces.

The field of cleaning/degreasing metal surfaces demands very good performance in particular as regards the degreasing power of the composition employed, and its non-foaming capacity. It should also be noted that such results are usually demanded even though the service conditions are very difficult. Spray methods encourage the appearance of foam because the treatment liquor is applied under pressure.

The compounds currently used in this field are mainly ethoxylated alkylphenols. Such compounds have highly satisfactory intrinsic degreasing properties. However, they suffer from a substantial disadvantage, namely encouraging foam formation. This undesirable phenomenon for this application is reduced by adding an antifoaming agent such as a linear alkoxyated alcohol. However, the presence of that type of compound reduces the degreasing activity of the ethoxylated alkylphenol in a manner that is not negligible and may be substantial. It is not possible to compensate for the loss of activity by adding sufficient additional alkylphenol to recover the initial level of activity.

International patent application WO-A-96/01245 describes alkoxyated terpene compounds presented as good degreasing agents that do not greatly encourage foam formation. The compounds of the examples are derivatives of nopol or arbanol type terpene compounds, and contain 0 to 2 oxypropylene moieties and 3.3 to 10.3 oxyethylene moieties.

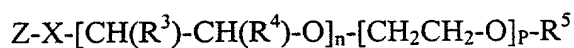
Such compounds, the importance of which is not questioned here, do not produce good results, however, in the very particular application of treating hard surfaces, in particular metal surfaces. They do not simultaneously satisfy the two criteria required in this field, namely a high degreasing activity and antifoaming action. This means that the amount of compound in the

formulation must be increased and/or an antifoaming agent has to be added, with known disadvantages. It is also stated that despite the addition of large quantities of such compounds, the degreasing levels achieved are at most equivalent to those of ethoxylated alkylphenols.

As can be seen, compounds with a very high degreasing capacity, which do not cause the appearance of a large amount of foam, must be available. The use of such compounds would have the double advantage of not making the use of large quantities of this compound necessary, and not requiring the addition of supplemental compounds such as antifoaming agents.

The aim of the present invention is to propose the use of a particular terpene compound that satisfies the double criteria cited above, in application to the cleaning/degreasing of hard surfaces, in particular metal surfaces, at low concentrations.

The present invention concerns the use, in cleaning/degreasing hard surfaces, of at least one compound employed in a concentration in the range 0.01 to 10 g/l during use of said compound, having the following formula:



in which formula:

- Z represents a bicyclo[a,b,c]heptenyl or bicyclo[a,b,c]heptyl radical, where:

$$a + b + c = 5$$

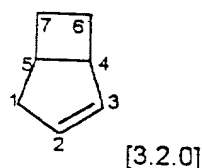
$$a = 2, 3 \text{ or } 4;$$

$$b = 2 \text{ or } 1;$$

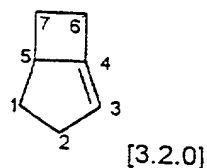
$$c = 0 \text{ or } 1;$$

said radical optionally being substituted by at least one C₁-C₆ alkyl radical and comprising a backbone Z selected from those indicated below, or the corresponding backbones minus the double bond:

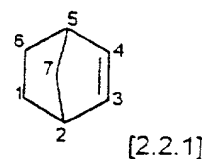
a)



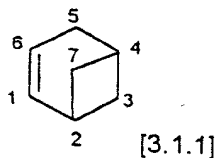
b)



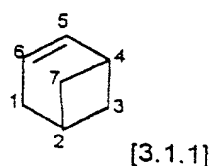
c)



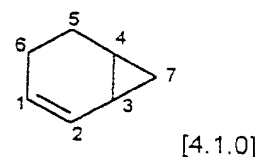
d)



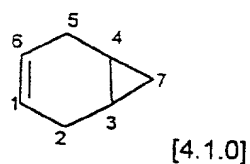
e)



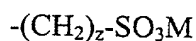
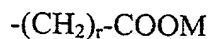
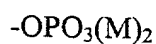
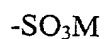
f)



g)



- X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ or $-\text{O}-\text{CH}(\text{R}'^1)-\text{CH}(\text{R}'^2)-\text{O}-$, in which:
 - R^1 , R^2 , R'^1 and R'^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical, preferably $\text{C}_1\text{-C}_6$;
 - R^3 and R^4 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical, provided that at least one of radicals R^3 or R^4 is other than hydrogen;
 - R^5 represents hydrogen, linear, branched or cyclic, saturated or unsaturated, aromatic or non aromatic $\text{C}_1\text{-C}_{22}$ hydrocarbon radical,, which may be substituted, or a group selected from the following:



in which formulae:

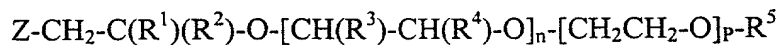
- M represents hydrogen, an alkali metal or an ammonium function $N(R)_4^+$, in which R, which may or may not be identical, represents hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1-C_{22} hydrocarbon radical,, which may be hydroxylated;
- r is in the range 1 to 6;
- z is in the range 1 to 6;
- n is a whole or fractional number in the range 3 to 5 inclusive;
- p is a whole or fractional number in the range 6 to 10, limits excluded.

Further advantages and characteristics of the present invention will become more clear from the following description and examples.

Firstly, the compounds employed in the present invention will be described.

A first type of compound is defined by formula (I), in which X is equal to
 $-CH_2-C(R^1)(R^2)-O-$.

This compound, hereinafter termed (Ia), corresponds to the following formula:



in which Z, R^1 , R^2 , R^3 , R^4 , R^5 , n and p have the meanings given above.

In one preferred implementation of the invention, radical Z is selected from radicals with formulas c) to g).

It should be noted that radical Z is more particularly attached to the remainder of the chain via one of carbon atoms 1 to 6; carbon atoms 1, 5 and 6 are preferred.

Further, radical Z can be substituted on at least one of its carbon atoms by two C_1-C_6 alkyl radicals, preferably two methyl radicals.

More particularly, the 7 carbon is substituted by these two alkyl radicals, more precisely two methyl radicals.

One preferred compound of the invention is, therefore, constituted by a compound in which radical Z corresponds to one of those appearing in figures c) to g), and more preferably radicals d) and e); radical Z being substituted by two methyl radicals located on the 7 carbon.

In a more advantageous implementation of the invention, radical Z corresponds to formulas d) or e), bonded to the remainder of the chain via the 5 or 1 carbon, and carrying two methyl substituents on the 7 carbon.

Preferably, R^1 , R^2 , which may or may not be identical, represent a hydrogen or a methyl radical. Preferably, R^1 , R^2 represent a hydrogen atom.

As indicated above, radicals R^3 and R^4 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1 - C_{22} hydrocarbon radical, provided that at least one of radicals R^3 or R^4 is other than hydrogen.

More precisely, said radicals represent hydrogen or a C_1 - C_6 alkyl radical, preferably the methyl radical or the ethyl radical, provided that at least one of these two radicals is other than hydrogen. Preferably, one of the radicals represents hydrogen, and the other represents a methyl radical.

R^5 represents hydrogen, a linear, branched or cyclic, saturated or unsaturated, aromatic or non aromatic C_1 - C_{22} hydrocarbon radical, which may be substituted, or a group selected from the following: $-SO_3M$, $-OPO_3(M)_2$, $-(CH_2)_r-COOM$, $-(CH_2)_z-SO_3M$, in which formulae:

- M represents hydrogen, an alkali metal or an ammonium function $N(R)_4^+$, in which R, which may or may not be identical, represents hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1 - C_{22} hydrocarbon radical,, which may be hydroxylated;
- r is in the range 1 to 6;
- z is in the range 1 to 6.

When R^5 is a hydrocarbon radical, this latter is more particularly a C_1 - C_6 alkyl radical or an alkylphenyl radical, optionally substituted by a halogen (for example chlorine).

Preferably, R^5 is a hydrogen atom.

Finally, n is a whole or fractional number in the range 3 to 5 inclusive and p is a whole or fractional number in the range 6 to 10, limits excluded.

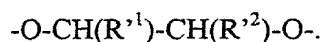
In a first particular implementation of the present invention, the value of n is 3.

Further, the value of p is more particularly in the range 6.2 to 7, limits included.
Preferably, p is in the range 6.3 to 7, limits included.

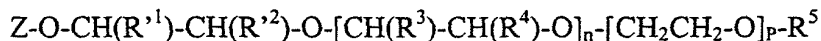
In a second particular implementation of the invention, n is in the range 4 to 5, limits included.

Further, the value of p is preferably in the range 7 inclusive to 10 exclusive, preferably in the range 8 inclusive to 10 exclusive.

A second type of compounds is defined by formula (I) in which X represents



Thus, this compound, hereinafter termed compound (Ib), corresponds to the following formula:



in which formula Z , R'^1 , R'^2 , R^3 , R^4 , R^5 , n and p have the meanings given above.

In one preferred implementation of the invention, radical Z corresponds to radical c), the bicyclic compound containing no double bond.

Here again, it should be noted that radical Z is more particularly attached to the remainder of the chain via one of carbon atoms 1 to 6; carbon atoms 1, 3, 4 or 6 are preferred.

Further, radical Z can be substituted on at least one of its carbon atoms by two C_1 - C_6 alkyl radicals, preferably two methyl radicals.

More particularly, the 7 carbon is substituted by these two alkyl radicals, more precisely two methyl radicals.

Further, carbon atoms 2 or 5 of radical Z carry a C_1 - C_6 substituent, preferably a methyl radical.

More particularly, and as mentioned above, radicals R'^1 and R'^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated C_1 - C_{22} hydrocarbon radical, provided that one of the two is other than hydrogen.

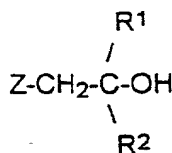
In a particular implementation of the invention, said radicals represent hydrogen or a C_1 - C_6 alkyl radical, preferably the methyl radical.

The foregoing regarding radicals R^3 , R^4 and R^5 and the values of n and p and the preferred variations concerning these values also apply in this case and will not be repeated.

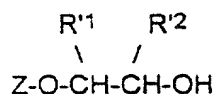
One particularly advantageous implementation of the invention is constituted by the use of compounds of type (Ia) described above.

The compounds of the invention can be prepared by reacting:

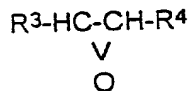
To obtain compounds (Ia), a reactant with formula (IIa):



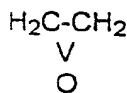
or to obtain compound (Ib), a reactant with formula (IIb):



with firstly, a reactant with formula (IIIop):



then, secondly, with a reactant with formula (IIIoe)



radicals Z , R^1 , R^2 , R^3 and R^4 having been defined above.

The reaction can be carried out in the presence of a catalyst.

Suitable catalysts that can be cited are strong bases such as alkali metal or alkaline-earth metal hydroxides or quaternary ammonium hydroxides of the $N(R)_4^+$ type in which R, which may or may not be identical, represents hydrogen or a C_1 - C_6 alkyl radical, preferably methyl or ethyl. Sodium, potassium and tetramethylammonium hydroxides are suitable for carrying out this reaction.

It is also possible to use catalysts selected from alkali metal or alkaline-earth metal alkoxides, such as sodium or potassium methylate, ethylate or teriobutylate. It should be noted that it is also possible to use primary, secondary or tertiary amines as the catalyst, preferably aliphatic amines, such amines possibly comprising other functions such as ether functions. An example of a catalyst of this type that can be cited is N,N-dimethylaurylamine.

In the case of a basic catalyst, the quantity is more particularly in the range 0.5 to 40 mg with respect to the final product weight.

It is possible to carry out this reaction in the presence of a Lewis acid such as BF_3 (gaseous or in solution in an ether), $SnCl_4$, or $SbCl_5$.

More particularly, the quantity of acid catalyst is in the range 0.1 to 10 mmoles per mole of reactant (IIa) or (IIb).

Contact is carried out at a temperature that is sufficient for the reaction to occur. By way of indication, the temperature is more than $100^\circ C$, more particularly in the range $120^\circ C$ to $250^\circ C$, and preferably in the range $150^\circ C$ to $200^\circ C$.

Advantageously, the reaction is carried out in an atmosphere that is inert under the reaction conditions, such as nitrogen, or a rare gas such as argon, or carbon monoxide. Nitrogen is preferred.

The reaction can be carried out at atmospheric pressure, under reduced pressure or under a slight over pressure. Normally, the pressure is in the range 1 to 4 bars.

The preparation of reactants (IIa) and (IIb) has been described in WO-A-96/01245, reference to which should be made.

The quantities of compounds (IIIop) and (IIIoe) are calculated as a function of the characteristics of formula (I), more particularly the desired values of n and p. These two compounds are introduced in succession, to obtain a block compound with formula (I).

At the end of the reaction, the reaction mixture is preferably neutralised to obtain a pH in the range 5 to 8, preferably 6 to 7.

Neutralisation is carried out using acetic acid, a hydroxide, sodium carbon or bicarbonate, depending on the nature of the catalyst used in the reaction.

At the end of this reaction, compound (I) is such that radical R^5 is hydrogen.

It is possible to carry out a functionalisation step of said radical, i.e., a step aimed at transforming the terminal hydrogen into another radical R^5 as defined above. An etherification or esterification operation can be carried out on the terminal hydrogen; this step is well known per se; it is preferably carried out after neutralisation.

The following can be prepared:

- ether sulphates ($R^5 = -SO_3M$), as described in British patent GB-A-1 111 208 or United States patent US-A-3 392 185;
- ether phosphates ($R^5 = -OPO_3(M)_2$), as described in US-A-3 331 896;
- ether carboxylates ($R^5 = -(CH_2)-COOM$), as described in US-A-2 623 900 or US-A-2 983 738;
- ether sulphonates ($R^5 = -(CH_2)_2-SO_3M$), as described in US-A-2 115 192, US-A-4 978 780 or K. SUGA, Austr. J. Chem., 21, 2333 (1968)
- alkyl ethers ($R^5 = \text{hydrocarbon radical}$) as described in US-A-2 913 416.

Further details regarding functionalisation modes are described in WO-A-96/01245.

The compounds of the invention are thus used as a degreasing agent for hard surfaces and more particularly for metal surfaces.

The compounds of the invention can be used in the metal treatment industry, and also in any application in which such surfaces are degreased, such as degreasing oil wells or oil production platforms.

As indicated above, the compound employed in accordance with the invention is used in a quantity in the range 0.01 to 10 g/l, at the time of use.

More particularly, when degreasing metal plates or sheets, the compound concentration is in the range 0.01 to 5 g/l during use.

In the oil production field, more particularly platform degreasing, the concentration of compounds is in the range 0.01 to 10 g/l at the time of use of compounds used in accordance with the invention.

With more particular regard to well cleaning, the compound of the invention is used in a concentration that is preferably in the range 0.01 to 5 g/l.

The aqueous degreasing or detergent media containing the compounds of the invention are preferably used at a temperature that is at least of the order of their cloud point.

The degreasing formulations for metal plates or sheets in an alkaline medium comprise, in addition to the compound of the invention, an alkaline lye comprising at least one of the following compounds:

- at least one pH control agent such as a hydroxide, carbonate, sesquicarbonate, or alkali metal or alkaline-earth metal bicarbonate;
- at least one polyphosphate such as a tripolyphosphate, pyrophosphate, orthophosphate or hexametaphosphate of an alkali metal, alkaline-earth metal, $N(R_4^+)$ type ammonium where R represents hydrogen, a C_1 - C_4 alkyl radical that may optionally contain an oxygen atom,;
- at least one alkali metal silicate such as alkali metal metasilicates, anhydrous or otherwise, or mixtures thereof.

The concentration of alkaline lye during use is in the range 1 to 100 g/l, more particularly in the range 5 to 20 g/l.

The formulations can also comprise:

- at least one hydrotropic electrolyte such as benzenesulphonates, mono- or di-alkyl (C₁-C₄) benzene sulphonates, toluene-, xylene- or cumene-sulphonates, alcohols or glycols;
- at least one sequestering agent such as nitriloacetic acid, ethylenediamine tetraacetic acid, ethylenediamine tetramethylphosphonic acid, nitrilotrimethylene phosphonic acid or salts thereof;
- buffer agents such as alkanolamines, ethylenediamine;
- metal corrosion inhibitors.

The concentrations of these compounds can lie within the normal ranges for this field.

However, by way of illustration, the amount of each of the compounds is from 0 to 1.5 g/l at the time of use.

In addition to the compound of the invention, aqueous compositions used to degrease oil platforms can comprise,:

- at least one hydrotropic electrolyte such as benzenesulphonates, mono- or di-alkyl (C₁-c₄) benzene sulphonates, toluene-, xylene- or cumene-sulphonates, alcohols and glycols;
- at least one pH control agent such as alkali metal carbonates, sesquicarbonates or bicarbonates;
- additives such as enzymes, metal corrosion inhibitors.

Here again, the concentrations of these compounds are within the normal range for the field. However, an illustrative concentration for each of the compounds is from 0 to 1.5 g/l at the time of use.

In addition to the compound of the invention, aqueous media employed to clean oil wells can comprise:

- at least one pH adjusting agent such as alkali metal carbonates, sesquicarbonates or bicarbonates, or alkali metal or alkaline-earth metal hydroxides;
- additives such as enzymes in an amount of up to 5% of the total aqueous composition weight, or metal corrosion inhibiting agents;
- if necessary, weighting agents to maintain a satisfactory hydrostatic pressure in the well.

Examples of such compounds that can be cited are soluble or at least partially soluble salts such as alkali metal or alkaline-earth metal halides. It is also possible to use alkali or alkaline-earth metal sulphates, carbonates, bicarbonates, silicates, or phosphates, used alone or as a mixture. It is also possible to use organic acid salts such as alkali metal or alkaline-earth metal formates or acetates. Although not preferred, it is possible to use insoluble salts such as alkaline-earth metal sulphates, silicates or carbonates; or alkaline-earth metal or zinc bromides;

- hydrocolloids such as polysaccharides of plant origin, such as polygalactomannanes, cellulose, starches and their derivatives; or polysaccharides of bacterial origin such as xanthan gum or deacetylated derivatives.

Although not preferred, the scope of the invention encompasses adding surfactants (anionic, cationic, zwitterionic, amphoteric), preferably low foaming surfactants, and other additives to the formulations described above.

Non-limiting examples illustrating the invention will now be given.

EXAMPLE 1

This example concerns the synthesis of 6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-ethanol containing differing numbers of oxypropylene (OP) units and oxyethylene (OE) units.

6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-ethanol comprising x oxypropylene (OP) units and y oxyethylene (OE) units was synthesised as follows:

the 6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-ethanol (nopol) used is sold by Fluka.

The nopol (6 moles) and an aqueous potassium hydroxide solution (50%, 3.1 g) were introduced into a 5 litre ethoxylation reactor.

The reaction medium was dehydrated at 120°C in a stream of nitrogen.

It was then heated to 170°C and propylene oxide (x molar equivalents) was introduced. Once the propylene oxide had been introduced, ethylene oxide was added (y molar equivalents).

The reaction medium was then cooled and neutralised by adding acetic acid to a pH of 7.

The liquid obtained was filtered through absorbent clay (Clarcel DIC).

Table of compounds synthesised using the method described above:

Compounds	Number of OP (x)	Number of OE (y)
Invention		
1	3	6.3
2	3	7
3	3	7.5
4	4	7
5	4	8
6	5	7
7	5	8
Comparative		
8	3	6
9	3	10
10	2	5

EXAMPLE 2

This example compares the degreasing properties of compounds 1 and 3 of the invention and comparative compounds 10, 11 and 12, and those obtained with defoamed Igepal NP10.

- Greasing

Pre-degreased "Q-Panel" Stock n° R-36 type "Dull matt finish" 0.8 x 76 x 152 mm steel plates were immersed for 2 minutes in a whole lubricating oil formulated for rolling steel sheets for automobiles, sold by Quaker Chemicals as QUAKER 6130 N, then suspended for 24 hours to drain.

- Cleaning and rinsing

The greased plates were introduced into a CIEMME LARO 350 degreasing spray machine. The degreasing temperature was 45°C and the pressure was 2 bars.

Before recording the degreasing quality, each face of the plates was rinsed for 5 seconds in a stream of water flowing at 2 ml/min.

- Degreasing medium

A basic degreasing medium (pH 12-13) was prepared by diluting 20 g/l of an aqueous lye containing 45% by weight of active substance constituted by equal weights of potassium hydroxide, sodium metasilicate (SIMET GA5, granules of anhydrous metasilicate and pentahydrated metasilicate sold by Rhodia Chimie) and tetrapotassium pyrophosphate and adding 1 g/l of test compound.

The results are shown in the table below:

Compound	Degreasing time (*)
1	30 seconds
3	30 seconds
8 (comparative)	45 seconds
9 (comparative)	55 seconds
10 (comparative)	> 2 minutes
Defoamed Igepal (**)	45 seconds

(*) The degreasing time is the time required to completely coat the two faces with a continuous film of water.

(**) Defoamed Igepal is a mixture of Igepal CO-660 (nonylphenol 10 OE) and Miravon B12DF (50/50).

Clearly, the product of the invention is more active than any of the agents employed for comparative purposes.

EXAMPLE 3

This example compares the degreasing properties of compounds 4 to 7 of the invention and those obtained with non-defoamed Igepal NP10.

- Greasing

An oil with trade name Pennzoil 80W-90 sold by Pennzoil Products Company (Houston, Texas) was applied to pre-degreased 0.8 x 24 x 101 mm steel plates using a brush to obtain an oil coating of about 0.015 g/cm². They were then stored in air for 24 hours.

5 The quantity of oil was measured by weighing the plate before and after application of the oil

- Cleaning and rinsing

The plate was immersed in one litre of degreasing medium, with magnetic stirring (100 rpm) at 50°C for 10 minutes.

10 Before reading the degreasing quality, each face of the plates was rinsed for 5 seconds in a stream of water flowing at 2 ml/min, then dried at 80°C.

The efficacy of the operation was calculated by weighing the plate before and after treatment with the degreasing medium.

- Degreasing medium

15 A basic degreasing medium (pH 12-13) was prepared by diluting 20 g/l of an aqueous lye containing 1.5 g/l of tetrapotassium pyrophosphate and adding 1 g/l of test compound.

The results are shown in the table below:

Compound	% of oil removed
4	58
5	62
6	54
7	72
Igepal (*)	51

(*)Igepal is Igepal CO-630, a nonylphenol 9 OE.

Clearly, the product of the invention is more active than the agents used for comparative purposes.

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EXAMPLE 4

This example compares the foaming properties of compounds obtained in Examples 1 to 9, with those obtained with comparative compounds, namely defoamed Igepal and Igepal alone.

Foam test:

A basic degreasing medium (pH 12-13) was prepared by diluting 20 g/l of an aqueous lye containing 45% by weight of active substance constituted by equal weights of potassium hydroxide, sodium metasilicate (SIMET GA5, granules of anhydrous metasilicate and pentahydrated metasilicate sold by Rhodia Chimie) and tetrapotassium pyrophosphate and adding 1 g/l of test compound.

The test was carried out at 50°C.

The foam was obtained by stirring 900 g of solution using a turbomixer (40 mm turbine, speed 2000 rpm) for 5 minutes.

The foam level was recorded for a sample after allowing to stand for 5 minutes.

The results are shown in the table below:

Compound	Foam level
1	20 ml
3	70 ml
4	20 ml
5	50 ml
6	20 ml
7	30 ml
Defoamed Igepal (*)	70 ml
Igepal alone (**)	950 ml

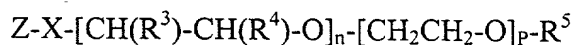
(*) Defoamed Igepal is a mixture of Igepal CO-660 (nonylphenol 10 OE) and Miravon B12DF (50/50).

(*)Igepal: Igepal CO-660 (nonylphenol 10 OE).

The above table, in combination with the degreasing results from the preceding examples, show that only the compounds of the invention satisfy the double criterion of high degreasing power and a foaming level compatible with use in degreasing metal plates.

CLAIMS

1. Use, in degreasing/ cleaning hard surfaces such as metal surfaces, of at least one compound employed in a concentration in the range 0.01 to 10 g/l during use of said compound, having the following formula (I):



in which formula:

- Z represents a bicyclo[a,b,c]heptenyl or bicyclo[a,b,c]heptyl radical, where:

$$a + b + c = 5$$

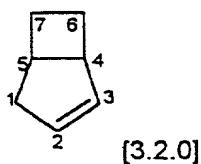
$$a = 2, 3 \text{ or } 4;$$

$$b = 2 \text{ or } 1;$$

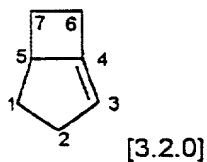
$$c = 0 \text{ or } 1;$$

said radical optionally being substituted by at least one $\text{C}_1\text{-C}_6$ alkyl radical and comprising a backbone Z selected from those indicated below, or the corresponding backbones minus the double bond:

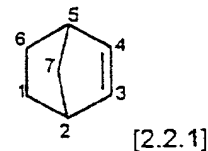
a)



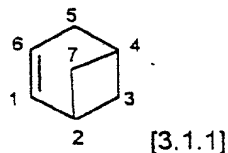
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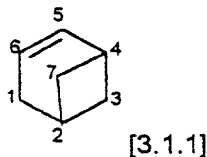
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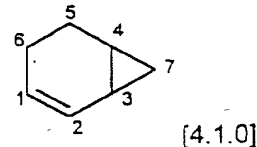
d)



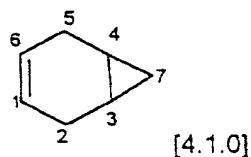
e)



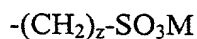
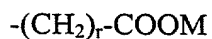
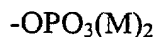
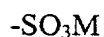
f)



g)



- X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ or $-\text{O}-\text{CH}(\text{R}'^1)-\text{CH}(\text{R}'^2)-\text{O}-$, in which:
 - $\text{R}^1, \text{R}^2, \text{R}'^1$ and R'^2 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical, preferably $\text{C}_1\text{-C}_6$;
 - R^3 and R^4 , which may be identical or different, represent hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical, provided that at least one of radicals R^3 or R^4 is other than hydrogen;
 - R^5 represents hydrogen, linear, branched or cyclic, saturated or unsaturated, aromatic or non-aromatic $\text{C}_1\text{-C}_{22}$ hydrocarbon radical,, which may be substituted, or a group selected from the following:



in which formulae:

- M represents hydrogen, an alkali metal or an ammonium function $\text{N}(\text{R})_4^+$, in which R, which may or may not be identical, represents hydrogen or a linear, branched or cyclic, saturated or unsaturated $\text{C}_1\text{-C}_{22}$ hydrocarbon radical,, which may be hydroxylated;
- r is in the range 1 to 6;
- z is in the range 1 to 6;
- n is a whole or fractional number in the range 3 to 5 inclusive;
- p is a whole or fractional number in the range 6 to 10, limits excluded.

2. Use according to the preceding claim, characterized in that the compound is such that in formula (I), n is equal to 3.
3. Use according to any one of the preceding claims, characterized in that the compound is such that in formula (I), p is in the range 6.2 to 7, limits included, preferably in the range 6.3 to 7, limits included.
4. Use according to claim 1, characterized in that the compound is such that in formula (I), n is in the range 4 to 5.
5. Use according to the preceding claim, characterized in that the compound is such that in formula (I), p is in the range 7 inclusive to 10 exclusive, preferably in the range 8 inclusive to 10 exclusive.
6. Use according to any one of the preceding claims, characterized in that the compound is such that in formula (I), radical Z is substituted on at least one of its carbon atoms by two C_1 - C_6 alkyl radicals.
7. Use according to any one of the preceding claims, characterized in that the compound is such that in formula (I), X represents $-\text{CH}_2-\text{C}(\text{R}^1)(\text{R}^2)-\text{O}-$ and in that the backbone Z is selected from formulae c) to g).
8. Use according to any one of the preceding claims, characterized in that the compound is such that in formula (I), backbone Z is selected from formulae d) and e).
9. Use according to any one of claims 1 to 5, characterized in that the compound is such that in formula (I), X represents $-\text{O}-\text{CH}(\text{R}'^1)-\text{C}(\text{R}'^2)-\text{O}-$ and in that radical Z corresponds to backbone c), the bicyclic backbone being free of a double bond.
10. Use according to the preceding claims, characterized in that the compound is such that in formula (I), radical Z is substituted by a C_1 - C_6 alkyl radical, preferably a methyl radical on carbon 2 or carbon 5 of the bicycle.

11. Use according to any one of the preceding claims, characterized in that the compound is used for degreasing/cleaning metal plates; the concentration of compound is in the range 0.01 to 5 g/l during use.
12. Use according to any one of claims 1 to 10, characterized in that the compound is used for degreasing/cleaning platforms; the concentration of compound is in the range 0.01 to 10 g/l during use.
13. Use according to any one of claims 1 to 10, characterized in that the compound is used for degreasing/cleaning oil production wells; the concentration of compound is in the range 0.01 to 5 g/l.

**USE OF POLYOXYPROPYLENE/POLYOXYETHYLENE TERPENE COMPOUNDS
AS DEGREASING AGENTS FOR HARD SURFACES**

Abstract of the Disclosure

The invention concerns the use in degreasing/cleansing applications of hard surfaces, such as metal surfaces, of at least a compound derived from a terpene and comprising a number of oxypropylene units ranging between 3 and 5, and a number of oxyethylene units ranging between 6 and 10, exclusively. The compound is provided with a concentration lower than 10 g/l, more particularly between 0.01 and 5 g/l, when used.

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COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(include Reference to PCT International Applications) PCT/FR00/02325

ATTORNEY'S DOCKET NO
RN99101

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

USE OF POLYOXYPROPYLENE/POLYOXYETHYLENE TERPENE COMPOUNDS AS DEGREASING AGENTS FOR HARD SURFACES

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application

Serial No. _____

on _____

and was amended

on _____ (if applicable)

☒ was filed as PCT international application

Number PCT/FR00/02325

on August 16, 2000,

and amended under PCT ARTICLE 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations. §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY PCT indicate PCT	APPLICATION NUMBER	DATE OF FILING (day month year)	PRIORITY CLAIMED UNDER 35 USC 119	
FRANCE	99/10559	17 August , 1999	<input checked="" type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Continued) (include Reference to PCT International Applications) PCT/FR00/02325				ATTORNEY'S DOCKET NO RN99101	
I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:					
PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120					
U.S. APPLICATIONS			STATUS (CHECK ONE)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED	
PCT APPLICATIONS DESIGNATING THE U.S.					
PCT APPLICATION NO	PCT FILING DATE	US SERIAL NUMBERS ASSIGNED (if any)			
POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney's and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (List name and registration number) JOHN A. SHEDDEN - Reg. No. 25,644, JOHN D. WOOD - Reg. No. 31,446 Kevin McVEIGH - Reg. No. 33,017					
Send Correspondence to: JOHN A. SHEDDEN INTELLECTUAL PROPERTY DEPT. RHODIA INC. 259 PROSPECT PLAINS ROAD, CN 7500, CRANBURY, NJ 08512-7500			Direct Telephone Calls to: <small>(name and telephone number)</small> JOHN A. SHEDDEN (609) 860-4190		
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	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY	
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	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY	
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203	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN	SECOND GIVEN NAME	
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP	
	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY	
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.					
SIGNATURE OF INVENTOR 201		SIGNATURE OF INVENTOR 202		SIGNATURE OF INVENTOR 203	
DATE 4.3.2002		DATE 4.03.2002		DATE	